Introducing of QC sample taken from internal process system in nuclear power plant when measuring boric acid

Abstract

Boron is determined by titration and the demand on within-lab reproducibility standard deviation is 0.2 % or 4 mg/kg (ppm) at a level of 2,000 mg/kg. Previously the QC sample for boron (Figure 1) titration has been prepared by weighing a given amount of boric acid (H_3BO_3 p.a. min 99.8%) and adding ultra pure water to a total weight of 5,000 g. After 2-3 hours of continuous stirring and heating to 70ºC, the heat was turned off but the stirring continued till the next day.

The prepared standard solution lasted about 1-2 month.

This method of control sample preparation was time consuming, difficult to prepare the exact concentration we wanted and had to be redone quite frequently.

The following describes the way to introducing QC sample taken from internal process system.

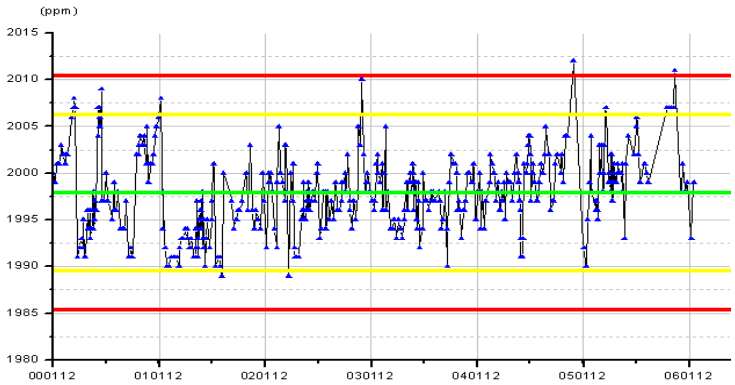

Figure 1. QC results for boron (from prepared standard solution) by titration analysed between 2000 and 2006. Each point represent a single (duplicate) measurement.

Previously - Prepared QC sample

Because of the high demand on the within-lab reproducibility standard deviation of the prepared standard solution (0.2%) we needed to prepare it with an expanded uncertainty less than 0,1 %. Many times we did not achieve this low uncertainty and the solution had to be poured out and prepared again. This caused a lot of irritation and was very time consuming.

Trying to improve the QC and to solve the problems with the QC-sample we found a solution in the "Handbook for Chemical Analytical Laboratories –Trollbook" (can be downloaded from www.nordtest.info TR569).

If the mean value of a new prepared standard solution for example wrongly prepared 2 ppm lower than wanted, a lot more results would be outside the WL compared with figure 1.

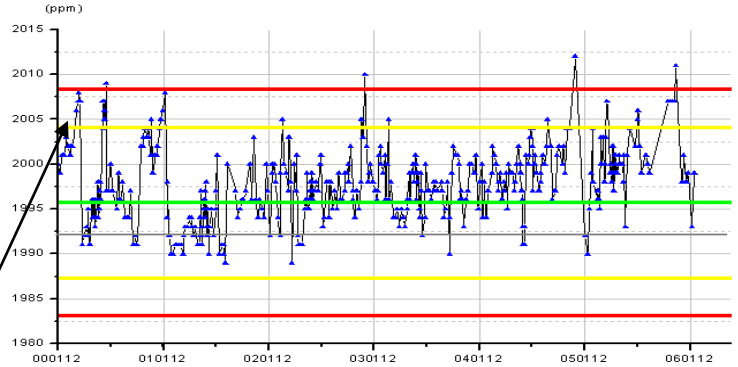

Figure 2. Example showing if the QC solution in figure 1 would have a 2 ppm lower mean value.

Now - QC sample from internal process system

During the last 2 years, process water from a boric acid tank has been used as QC sample (Figure 3). The concentration of boron in the tank varies over the year only slightly - approximately between 2,025 ppm and 2,080 ppm.

Each time a new batch (witch lasts approximately 6 month) is collected from the tank a new central line (3 measurements for temporary setting of the CL and after 20 measurements fixating the CL) is calculated. The control limits are based on standard deviation obtained previous years.

The control sample is measured as duplicates together with the daily samples at each time of analyses.

In order to further control the status of the method and instrument a certified reference material is analysed on a regularly basis.

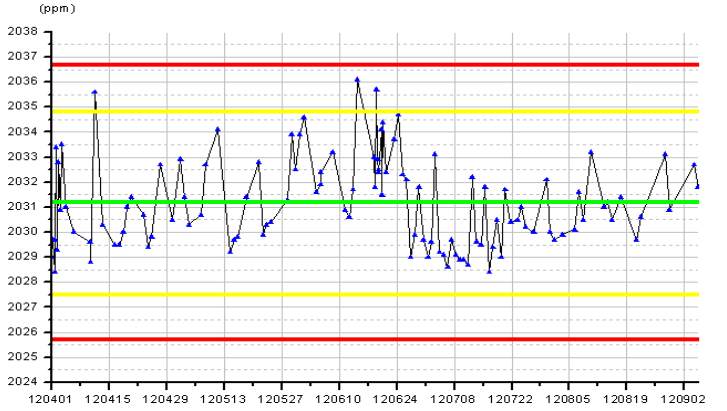

Figure 3. QC results for boron by titration (from one batch of QC sample) analysed from Jan to Sep 2012. Each point represent a single (duplicate) measurement.

Conclusion

- Using control samples from process system has facilitated the quality control considerably and the procedure is satisfactorily applied since the last 2 years.
- Instead of preparing a control sample which takes days and may have to be redone, using a control sample taken from process systems saves both time and money!

VATTENFALL